NATIONAL BUREAU OF STANDARDS REPORT

8959

Progress Report
on
Hardening Shrinkage Forces in Direct Filling Materials

U.S. DEPARTMENT OF COMMERCE
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Hardening Shrinkage Forces in Direct Filling Materials

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Hardening Shrinkage Forces in Direct Filling Materials

Abstract

Tensile forces develop when dental filling materials harden within a cavity, if there is bonding to the cavity walls. A test method was devised by which these forces could be measured. This was done by observing the loading required to maintain constant distance between two opposing walls (having known area) of an artificial cavity. If several broad assumptions are made it can be estimated that a tensile stress of at least 49 kg/cm^2 (700 psi) at the walls of a cavity may develop during the hardening of a direct filling resin. The corresponding stress for a silicate cement would be about 35 kg/cm^2 (500 psi). Other direct filling materials were also investigated. The magnitude of the stress depended on the exact test method, the material, and other factors.

The strength of adhesive bonding between a direct filling material and the cavity walls of a tooth must exceed the tensile stresses that develop during the hardening of the filling material, if the bonding is to remain intact.

1. Introduction

For a dental restoration to fill a cavity and retain close adaptation to the cavity walls, the adhesion or bond strength between the filling material and the tooth surfaces must at all times be at least as great as the tensile or shear forces tending to separate these materials. These disruptive forces arise, among other things, from differences in the thermal expansivities of the two materials, [1-4] from externally applied loads (e.g., during mastication), and possibly from volumetric shrinkage which may occur during the hardening of the filling material [5].

The fact that fillings placed in extracted teeth show percolation [6] (fluid exchange at the margins of the fillings with change in temperature) indicates that at least part of the interface can be disrupted without the aid of masticatory forces. There are numerous references in the literature to penetration of radioactive ions around fillings in extracted teeth immersed in water, indicating that the contiguity of the interface is incomplete [7-9]. In addition to flaws that may be formed during the placement of the material, marginal defects could conceivably occur as a result of displacement by water, thermal stresses, or tensile stressed due to hardening shrinkage.

The purpose of the present work is to determine if hardening forces develop tensile stresses, and if they do, to obtain an estimate of their order of magnitude. If the walls of a tooth cavity are wetted by a filling material and there is good cavity adaptation, then a material's volumetric shrinkage on hardening [10,11] would be expected to produce tensile stress at the material-tooth interface. This stress must be resisted by adhesion between the materials if the restoration is to remain hermetically sealed.

An estimate of the magnitude of the tensile stresses caused by the hardening of the filling material will assist in evaluating the results on tensile bond strengths that have been reported previously [12] and may suggest means of obtaining improved bonding.

2. Materials and Methods

Both commercial and experimental materials were used. The commercial materials are listed in Table 1. The experimental composites consisted of mixtures of a treated silica powder (Table 2) with four different monomer formulations. The monomers each contained a crosslinking methacrylate, (synthesized by the addition of methacrylic acid to the diglycidyl ether of Bisphenol A*), [13] thinned with methyl methacrylate or other methacrylate esters; a polymerization accelerator, such as N,N-dimethyl-p-toluidine; stabilizers, and other minor ingredients. The detailed description of such formulations is beyond the scope of this paper; information on such compositions is to be reported as a separate publication.

* D.E.R. 332, The Dow Chemical Company

The commercial materials were mixed according to the manufacturer's recommendations. The experimental materials were mixed on glass with a stellite spatula in the same manner as the mixing of silicate cement. The experiments were conducted in a room maintained at a temperature of 23.0 ± 2.0°C (73.4 ± 3.6°F) and a relative humidity between 40 and 60 percent.

Five test methods were used. It should be emphasized that in the first method, the "cavity walls" were not held at a constant distance apart (there was unknown strain), whereas in the other methods, the dimensions were essentially constant. The latter methods differed in "matrix materials" and in the manner of filling the cavities.

Method I. The materials were mixed as quickly as possible and placed into a tube that formed a short, cylindrical "cavity." The tube was of soft poly(ethylene) so that the materials would not adhere to it. The tube played the part of a "dental matrix." The end walls of the cavity were the ends of two stainless steel plungers (one of these plungers has been illustrated previously [12]. Small, shallow, dovetail depressions assisted in maintaining "adhesion" to the metallic end walls.

The steel plungers were parts of larger cylinders [12] which fitted with close tolerances into a stainless steel tube which held the plungers in alignment. The assembly was mounted vertically between the crosshead and a tensile load cell of a testing machine.*

As the material shrank on hardening, the response of the load cell was recorded automatically. The stress due to hardening shrinkage was defined as the maximum force that developed, divided by the cross-sectional area of the specimen, which was of the same area as that of the end walls of the cavity. The diameter of the cavity and each of the circular end walls was 6 mm (0.236 in), with an area of about 28.77 mm^2 (0.0438 square inch). The length of the cavity was varied and will be discussed later. In Method I, correction was not made for the compliance of the strain gauge (the cell deflection was stated by the manufacturer to be 0.00004 inch per pound) or for the yielding of the metallic parts of the assembly and connectors, which would lower the apparent stress values.

After each specimen had developed its maximum hardening shrinkage stress, the tensile load on the specimen was increased and the stress required to break the specimen loose from the end walls was recorded. Generally, this latter stress was appreciably higher than the apparent shrinkage stress, indicating that the bonding between the filling material and the end walls was adequate.

Method II. An optical strain gauge was placed on the assembly in such a way that dimensional changs of about 0.5µ (0.02 mil) between the steel ends of the cavity could be observed in Method II. By the use of the strain gauge and manual adjustments of the crosshead, the original distance between these "cavity walls" was kept very nearly constant. Otherwise, Method II was the same as Method I.

Method III. The filling material was placed in the cavity by a paint-in technic in Method III. The increments of direct filling resin, applied with a fine (000) sable brush, were placed through a small hole in the plastic tube "matrix." This allowed the optical strain gauge to be in position throughout the filling operation. Although it was not taken advantage of in these experiments, this method would allow controlled and predetermined specimen length. Time was not allowed for each increment to harden before the next increment was placed in the cavity. The hole in the matrix was covered with tin foil after the cavity had been filled. Otherwise, Method III was the same as Method II.

Method IV. Method IV was the same as Method II except that the soft plastic matrix tube was replaced by a surface-treated soda-lime-silica glass tube. The surface treatment consisted of wetting the glass with a 0.5 percent aqueous solution of 3-(trimethoxysilyl) propyl methacrylate**(the silane had been hydrolyzed with the aid of 0.1 percent of acetic acid per weight of solution), drying the glass, and then heating it to 116°C (240°F) for 20 minutes. The tube had a close, sliding fit over the stainless steel plungers, as had the plastic matrix.

* Instron, Type T T-C-L

** Z-6030, Dowing Corning Corp.

The surface treatment of the glass made it very adherent to the hardening resin, which, together with its stiffness, made it constrain the flow of the resin. A new tube had to be used for each measurement because of the bonding between the hardened resin and the treated glass surface.

Method V. Measurements were also made in which no matrix was used. The filling material was simply placed between the steel end walls of the plungers. There were some excesses and/or deficiencies in the peripheral areas with this method, but care was taken in attempting to keep the average cross-sectional area of the material the same as it was when a matrix tube was used. Otherwise, Method V was the same as Method II.

The resumé of the five test methods is given in Table 3.

In three cases (one each in Methods II, III, and IV), an iron-constant thermocouple was prepositioned in the cavity, and the temperature within the hardening material was recorded by an automatic recording potentiometer. The recording charts were synchronized so that the temperature within the hardening material could be compared with the stress acting on the cavity walls.

3. Results and Discussion

Measurable tensile forces developed during the hardening of all of the materials and with each of the methods.

Quantitatively, these hardening shrinkage forces varied with the test methods, with the kind of material under observation, and possibly with the "cavity dimensions" and other factors. The data on individual specimens are given in Tables 4 to 8. The values were calculated in pounds per square inch, rounded, and then converted to kilograms per square centimeter.

With Methods II, IV and V, the average time, which elapsed from the starting of the mix to the time when the fiducial reading of cavity dimensions was made, was 2.5 minutes; the range was 1.5 to 4.4 minutes. In only two cases, indicated in Table 8 (Method IV), were there indications by movements of the strain and/or stress indicators that the stress had started up before the fiducial readings were made at about four minutes. In these two cases, the final stress values may have been somewhat higher if the control of constant "cavity dimensions" (strain) had been initiated sooner.

Excess mixed material was manipulated as it and its counterpart in the assembly hardened. Qualitatively, the stress started to build up in the assembly only after the excess material had developed a notable degree of resistance to flow. With the resins measured, most of the stress increase followed the exothermic peak temperature, which corresponds to a relatively well-hardened material.

Since the maximum stresses took from about two to seven hours to develop in the direct filling resins, only one or two specimens could be measured per day, and, considering the number of variables, there are insufficient data to determine if any significant differences exist among the various commercial brands of similar materials. For this reason, the brands are given in letter codes which do not correspond to the alphabetical listing in Table 1. In the experimental composites (Table 6), the maximum stresses developed in about 0.3 to 2 hours after the start of the mixing.

When tested by Method II, the maximum hardening stresses of the materials decreased in this order: resin cement and direct filling resin, silicate cement, zinc phosphate cement, zinc oxide rosin-eugenol and silver amalgam. The silver amalgam developed a very slight maximum tensile stress (at about 15 to 30 minutes from the start of trituration) before the expansion of the material allowed the tensile stress to return to zero (at about 1 to 2 hours after trituration).

With the direct filling resins, Method IV gave highest stresses and Method I gave the lowest.

The most notable effect of the paint-in technic (Method III) was a retarding of the onset of stress build-up, probably as a result of the inhibiting (or retarding of polymerization due to the increased exposure to oxygen of the air. Also, unfortunately, air bubbles were trapped in each of these specimens.

In Method I (Table 4), the higher stresses appear to be associated with the longer specimens. With Method II, (Table 5) the two highest stresses were observed with the shortest specimens, in spite of the probability that they would have a lower exothermic temperature rise. The highest stresses of all were obtained with the treated glass

tubes (Method IV). The polymer adhered to the rigid glass tube and was thus restrained from flowing ("relaxation").

The experimental composites had stresses below 28 kg/cm^2 (400 psi), (Table 6), as compared with the direct filling resins which all had values higher than this when also tested by Method II. An intermediate range, which overlapped these, resulted from the use of a silica powder (Powder B, which is described in Table 2) with the monomer and catalyst of direct filling resin A (Table 6).

It was noted in Method IV that the highest stress, 134 kg/cm^2 (1,900 psi), was obtained with no "free area," the steel and the treated glass tube to which the resin adhered, completely encasing the hardening resin. The next lesser value of 117 kg/cm^2 (1,660 psi) was obtained with a window cut into the glass tube, giving some "free area" from which the resin could flow as it contracted. A smaller stress resulted when there was both a small window in the glass and trapped air bubbles within the resin. When nearly half of the diameter of the treated glass tube was cut away, forming a larger window or "free area," the remaining glass "side" imploded, due to the tensile stress of the adhering resin, when the stress on the steel end walls had reached 73 kg/cm^2 (1,040 psi). After the implosion, the stress dropped to 65 kg/cm^2 (920 psi) and then slowly increased to 72 kg/cm^2 (1,020 psi).

In the few cases of direct filling resins (using Methods II and V) having specimen lengths about 1 mm or less and containing no large air bubbles (Tables 5 and 8), the stresses were higher than the corresponding cases having lower "bound area" to "free air" ratios.

Air bubbles, large or small, visible or unseen, may have reduced the stress by providing "free areas" from which resin could flow. If so, this might ammount for some of the scatter in the data.

In Methods II, III and V, the direct filling resins with specimen lengths of more than about 1 mm developed stresses between about 28 kg/cm^2 (400 psi) and 59 kg/cm^2 (840 psi).

Comparison of the results of Method I with Method II has indicated that if the cavity walls can yield even slightly, the maximum tensile stress that develops is less. Due to their high moduli of elasticity, enamel and dentin would not be expected to yield very much, but, since their moduli are finite, there might be lessening of stress due to compliance of the cavity walls.

In the three cases where the temperature within the direct filling resin was plotted against the polymerization shrinkage stress, the onset of stress build-up appeared to precede slightly (0.3, 1 and 2 minutes, respectively) the exothermic peak temperature. It should not be assumed, therefore, that all of the tensile stress was due to "cooling shrinkage" (i.e., a function of the thermal expansivity of the material). The application of external heat (later), sufficient to rewarm the three specimens to their peak temperatures, did not reduce the stresses to zero.

For the objective of obtaining low hardening stresses, use of polymer powders or fillers with liquids containing mostly methyl methacrylate does not appear promising, due to the high volumetric polymerization shrinkage of this monomer. However, the use of organic liquids, having relatively low polymerization shrinkage, with treated fused silica fillers does appear to reduce volumetric shrinkage [11] and hardening shrinkage stresses.

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Table 1.

Commercial Materials Tested For Hardening Shrinkage Forces

Material	Source
Bonfil	The L. D. Caulk Co.
Fast Crown	Acralite Co., Inc.
Grip Cement	The L. D. Caulk Co.
Kadon	The L. D. Caulk Co.
Mer-Don 7	American Consolidated Mfg. Co., Inc.
Moyco Z.R.E. Zinc Oxide Rosin-Eugenol Cement	The J. Bird Moyer Co., Inc.
Improved Replica Color-fast Plastic Filling Material	Cosmos Dental Products, Inc.
Sevriton	The Amalgamated Dental Co., Ltd.
Sevriton Simplified	The Amalgamated Dental Co., Ltd.
S. S. White New Filling Procelain	The S. S. White Dental Mfg. Co.
S. S. White Zinc Cement Improved	The S. S. White Dental Mfg. Co.
Texton	The S. S. White Dental Mfg. Co.
A silver amalgam:	
(alloy: True Dentalloy, Cut A)	The S. S. White Dental Mfg. Co.
(mercury: American Mercury)	Pure-Lab Co. of America

Table 2

Experimental Materials Tested For Stress From Hardening Shrinkage; Composition of Silica Powder A

Powder	Estimated particle size (μ)	Bulk volume of treated powder (%)	Weight (%)	Glass Density (g/cc)	Coupling agent used* (%)	Powder Source
Cataflex "202" Sign Beads (X-ray opaque Ba-Ti glass)	44-53	33	57†	4.1	0.27	Cataphote Corp.
Spheriodal fused silica‡	5-37	33	27	2.2	1.0	Processed by TAFA Div. of Humphreys Corp.
Min-U-Sil (ground quartz)	<0.5-5	24	10	2.65	2.5	Pennsylvania Glass Sand Corp.
Cab-O-Sil M-5 (pyrogenic silica)	>0.015	10	5	2.2	50	Cabot Corp.
Benzoyl peroxide	-	-	1.0	-	-	Fischer Scientific

* Each powder was treated separately with the given weight percent of 3-(trimethoxysilyl) propyl methacrylate (Z-6030, Dow Corning Corp.).

† Approximately 33 percent of this powder would be sufficient to match the X-ray opacity of hard tooth tissues.

‡ This powder, used alone, will be referred to in the text and in Table 3 as "Powder B".

Table 3

Résumé of Test Methods

Method	Specimen length or distance between "cavity walls"	"Matrix material"	Manner of mixing and filling cavity
I	Decreased during test	Soft tube	Bulk mix; packed in
II	Constant	Soft tube	Bulk mix; packed in
III	Constant	Soft tube	Paint-in
IV	Constant	Treated (adhesive) glass tube	Bulk mix; packed in
V	Constant	None	Bulk mix; packed in

Table 4

Stress from Hardening Shrinkage
in Direct Filling Materials

Determined by Method I

Material	Maximum stress developed (kg/cm²)	(psi	Specimen length (mm)
Direct filling resin A	6.5	93	1.17
	7.7	110	4.70
	8.4	120	9.62
	9.1	130	11.9
The monomer and catalyst of the direct filling resin A, used with Powder B*	5.3	75	1.27
	17.6	250	5.85
	17.6	250	10.5
Direct filling resin B	5.4	77	1.4
	11.3	160	6.06
	14.8	210	8.55
	16.9	240	12.
	21.8	310	10.8

* Powder B is treated, spheriodal, fused silica described in Table 2.

Table 5

Stress from Hardening Shrinkage
in Proprietary Direct Filling Resins

Determined by Method II

Material	Maximum stress developed (kg/cm²)		(psi		Specimen length (mm)
Direct filling resin A	(28)*	29	(400)*	410	1.39
		29		410	3.13
		34		480	1.88
	(41)	41	(580)	580	1.57
Direct filling resin B		30		420	5.8
		32		450	5.7
		37		530	4.84
		37		530	10.1
		39		550	13.8
	(32)	40	(450)	570	4.7
		40		570	8.5
		42		590	3.10
	(50)	56	(710)	800	2.6
		68		970	0.68
		89		1,270	1.04
Direct filling resin C		39		550	4.20

* The stress values in parentheses represent the stresses in specimens containing bubbles with no corrections made for the reduced cross-sectional areas.

Table 6

Stress from Hardening Shrinkage in Experimental Direct Filling Composites Prepared with Silica Powder A and Various Monomers

Determined by Method II

Monomers used	Maximum stress developed (kg/cm^2)	(psi)	Specimen length (mm)
	20	290	(2.1)*
The monomer and catalyst of direct filling resin A	27	390	1.85
	29	410	2.05
	46	650	(1.9)
Experimental liquids	0.4	6	1.4
	1.3	18	1.73
	2.1	30	1.94
	2.8	40	1.94
	7.0	100	1.77
	7.0	100	2.14
	11	160	2.54
	12	170	1.79
	16	230	2.53
	17	240	2.31
	20	290	1.97
	23	320	2.27
	23	330	2.4
	25	350	3.26

* The specimen-length values in parentheses are estimated values based on measurements made while specimens were within the assembly. Others were from measurements made directly on specimens removed intact from assembly.

Table 7

Stress from Hardening Shrinkage in Direct Filling Materials

Determined by Method II

Material	Maximum stress developed (kg/cm^2)	Maximum stress developed (psi)	Specimen length (mm)
	28	400	2.82
A silicate cement	30	430	2.05
	37	530	3.35
	39	560	2.98
	40	570	2.22
	15	210	2.1
A zinc phosphate cement	16	230	4.30
	17	240	3.19
	25	360	3.10
	26	370	2.35
	(52)* 54	(740)* 770	2.46
A resin cement	58	820	2.95
	(56) 62	(800) 880	1.95
	(49) 64	(690) 910	2.9
	0.5	7	1.65
A zinc oxide rosin-eugenol cement	1.2?	17?	2.9
	1.3	18	2.79
	1.5	21	2.6
A silver amalgam	0.4	6	1.26
	0.7	10	0.5
	1.7	24	2.98

* The stress values in parentheses represent the stresses in specimens containing bubbles with no corrections made for the reduced cross-sectional areas.

Table 8

Stress from Hardening Shrinkage in Proprietary Direct Filling Resins

Determined by Methods III, IV and V

Material	Method	Maximum stress developed (kg/cm^2)	(psi	Specimen length (mm)
Direct filling resin B	III (paint-in)	(34)* 39	(490)* 550	4.1
		(25) 52	(350) 740	(3.?)†
		(54) 57	(770) 810	1.8
		(56) 60	(790) 850	1.47
Direct filling resin B	IV (treated glass tube)	>73‡δ	>1,040‡δ	5.37
		(69) 90	(980)1,280	4.35
		117	1,660	2.0
		134δ	1,900δ	6.55
Direct filling resin				
F	V (no matrix)	(30) 32	(420) 450	0.62
A		(15) 34	(210) 480	1.87
C		39	550	1.10
E		(36) 34	(510) 630	0.62
B		54	770	1.75
H		59	840	1.5
G		61	870	1.0
D		70	990	0.5

* The stress values in parentheses represent the stresses in specimens containing bubbles with no corrections made for the reduced cross-sectional areas.

† The specimen-length values in parenthesis are estimated values based on measurements made while specimens were within the assembly. Others were from measurements made directly on specimens removed intact from assembly.

‡ The treated glass tube imploded when the tensile stress on the steel walls had reached the value given.

δ In these two measurements, the stress and strain had started to build up before the strain (cavity dimension) was first registered and thereafter kept constant; therefore, these two reported values may be somewhat lower than they should be.